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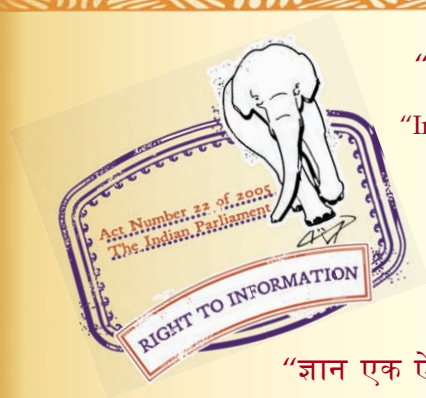
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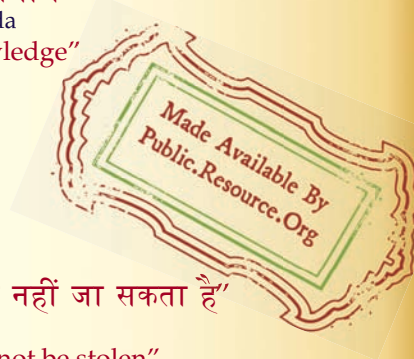
IS 5328 (1969): Method of test for determination of chemical composition of asbestos fibre [CED 53: Cement Matrix Products]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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Indian Standard

METHOD OF TEST FOR DETERMINATION OF CHEMICAL COMPOSITION OF ASBESTOS FIBRE

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

METHOD OF TEST FOR DETERMINATION OF CHEMICAL COMPOSITION OF ASBESTOS FIBRE

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Indian Standard

METHOD OF TEST FOR DETERMINATION OF CHEMICAL COMPOSITION OF ASBESTOS FIBRE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 13 August 1969, after the draft finalized by the Cement and Concrete Sectional Committee had been approved by the Civil Engineering Division Council.

0.2 A series of Indian Standards on testing procedures of asbestos fibre is being formulated so as to provide standard methods for obtaining physical and chemical properties of asbestos fibre which is used for manufacturing various asbestos cement (AC) products like AC sheets, AC pipes, etc. These testing procedures will be useful both for mine owners and the manufacturers of AC products provided they have the facilities to make these tests with reasonable accuracy and the personnel with the required degree of laboratory experience.

0.3 In the formulation of this standard due weightage has been given to international co-ordination among the standards and practices prevailing in different countries in addition to relating it to the practices in the field in this country. This has been met by basing the standard on 'Testing procedure for chrysotile asbestos fibre' jointly published by American Textile Institute, Mineral Fibre Products Bureau and Quebec Asbestos Mining Association.

0.4 This standard is one of the three Indian Standards on testing of asbestos fibre published so far. IS:3632-1969* and IS:4844-1968† are the other two standards published under this series.

0.5 In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS:2-1960‡.

*Method of test for determination of wet volume of asbestos fibre.

†Method of sampling and preparation of asbestos fibre for laboratory test purposes.

‡Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard covers the methods for the quantitative analysis for (a) silica, (b) ferrous oxide, (c) ferric oxide, (d) alumina, (e) calcium oxide, (f) magnesium oxide, (g) sodium oxide, (h) carbonates, and (j) water of crystallization in asbestos fibre.

2. OUTLINE OF METHOD

2.1 The samples are treated as silicates not decomposed by acids and are, therefore, fused with sodium carbonate to convert to soluble sodium salts. From the hydrochloric acid solution of the melt, silica is first removed by dehydration and precipitation. The filtrate containing iron and aluminium plus calcium and magnesium is treated with bromine water* to oxidize the iron to the ferric state and the latter together with the aluminium is precipitated with ammonia and ignited. To differentiate between ferric oxide and alumina, the ignited residue is fused with potassium pyrosulphate and the iron in the melt determined volumetrically. Another aliquot portion of the solution of the melt is used for determination of alumina by use of 8-hydroxyquinoline. The filtrate resulting from the ferric oxide and alumina precipitation contains calcium (if present) and magnesium. The calcium is precipitated from this filtrate, after the latter is acidified, as calcium oxalate. The latter is ignited and weighed as calcium oxide.

2.2 The filtrate after removal of the calcium is acidified with concentrated nitric acid and evaporated to dryness to ensure removal of ammonium salts as the latter will prevent complete precipitation of the magnesium. The residue is dissolved in dilute hydrochloric acid and the magnesium precipitated with ammonium-hydrogen phosphate in ammoniacal solution. The precipitate is carefully ignited and weighed as magnesium pyrophosphate. The water of crystallization is determined by ignition loss and corrected for carbonate content.

3. SAMPLING

3.1 The sample shall be the laboratory test sample in accordance with IS:4844-1968†.

4. PREPARATION OF LABORATORY TEST SPECIMEN

4.1 Quarter the laboratory sample down to approximately 25 g. Divide the 25 g sample into ten approximately equal portions and sub-divide

*If bromine water is not available, the oxidation can be affected with a few drops of concentrated nitric acid or hydrogen peroxide.

†Method of sampling and preparation of asbestos fibre for laboratory test purposes.

these portions into halves. Combine a half of each of the ten portions and mix thoroughly. Repeat this procedure until the sample weight is reduced to approximately 3 g. On a smooth and clean area spread the 3 g sample into a thin layer over an area of approximately 20×20 cm, in such a manner that pinches may be taken from all parts. Extract with tweezers five bundles of long fibre free from rock, grit or dust, each weighing about 0.5 g. Spread into a thin layer and extract at least five bundles of the fibre from different parts so that when these are combined and dried to constant weight at 110°C the test specimen will weigh 0.5 ± 0.02 g.

5. PREPARATION OF SAMPLE

5.1 Apparatus

5.1.1 *Agate Mortar and Pestle* — 125 ml capacity.

5.1.2 *Platinum Crucible* — 30 ml capacity.

5.1.3 *Oven* — 110°C .

5.1.4 *Electric Furnace* — $925^{\circ} \pm 25^{\circ}\text{C}$.

5.1.5 *Chemical Balance*

5.1.6 *Porcelain Crucible* — 20 ml capacity.

5.1.7 *Camel Hair Brush*

5.1.8 *Desiccator*

5.2 Reagents

5.2.1 *Sodium Carbonate* — solid.

5.3 Procedure—The test specimen is placed in a tared porcelain crucible and after being brought to constant weight at 110°C is cooled in a desiccator and weighed on a sensitive balance to the fourth decimal place. This weight minus the tare is the sample weight (W) for (a) silica, (b) ferric oxide, (c) alumina, (d) calcium oxide, and (e) magnesium oxide. Different samples shall be used for carbonates and water of crystallization. The crucible with specimen is then placed in a furnace at $925^{\circ} \pm 25^{\circ}\text{C}$ for one hour. After cooling, the specimen is reduced to as fine a mesh as possible with an agate mortar and pestle. About 3.0 g of sodium carbonate are added and ground intimately with the specimen for an intimate mixture. The contents of the mortar are then transferred to a 30 ml capacity platinum crucible using a camel hair brush to sweep the last of the mixture from the mortar. Successive small amounts of additional sodium carbonate are then ground in the mortar and transferred in like manner to the platinum crucible, to ensure removing of

the last trace of specimen from mortar to the crucible. Then sprinkle a little additional sodium carbonate on top of the intimate mix of powdered specimen and the sodium carbonate. Place a platinum lid on the crucible.

6. DETERMINATION OF SILICA BY THE HYDROFLUORIC ACID (GRAVIMETRIC) METHOD

6.1 Apparatus

6.1.1 *Platinum Crucible*

6.1.2 *Platinum Wire*

6.1.3 *Casserole* — 300 ml.

6.1.4 *Glass Triangle*

6.1.5 *Vented Chamber* — 110°C.

6.1.6 *Filter Paper* — Whatman No. 40.

6.1.7 *Water-Bath*

6.1.8 *Meker Burner*

6.1.9 *Vented Hood*

6.2 Reagents

6.2.1 *Dilute Hydrochloric Acid* — 2 N and 6 N.

6.2.2 *Concentrated Hydrochloric Acid* — specific gravity 1.16.

6.2.3 *Concentrated Sulphuric Acid* — specific gravity 1.84.

6.2.4 *Hydrofluoric Acid* — 40 percent.

6.3 Procedure

6.3.1 Heat the covered platinum crucible containing the intimate mix of powdered specimen and flux at first over a small flame to drive out any moisture present. Gradually raise the temperature until the highest heat of a good Meker burner is obtained. As soon as the mass melts quietly and there is no further evolution of carbon dioxide, the decomposition is complete. ☞

6.3.2 Wind a piece of platinum wire into a spiral and insert it into the fused mass. Remove the flame, allow the crucible to cool in the air somewhat and then play a stream of water upon the outside of the crucible. As soon as the crucible does not hiss when the water strikes it, quickly introduce enough water into the crucible to cover the melt. After about a minute carefully pull on the wire; usually the melt can be withdrawn from the crucible. If it does not come out easily, it can often be loosened by carefully heating the crucible.

6.3.3 Place the melt in a 300 ml casserole, add 25 ml of water, cover the casserole and carefully add 25 ml of 6 N hydrochloric acid. A lively evolution of carbon dioxide at once takes place, but as the silicic acid separates, the inner part of the cake gradually becomes coated with a film of silicic acid which protects it from the further action of the acid. Consequently, it is necessary to break up the cake from time to time by means of glass rod, until finally there is no evolution of gas and no more hard lumps remain.

6.3.4 After the evolution of carbon dioxide has nearly ceased, wash off the under side of the watch-glass, raise it by placing a glass triangle under it, and evaporate to dryness. Heat the residue for at least an hour at 110°C to dehydrate the silica.

6.3.5 Moisten the dry powder with concentrated hydrochloric acid (specific gravity 1.16) and allow the covered dish to stand 10 minutes at the ordinary temperature in order that the basic salts and oxides formed during the evaporation and drying may once more be changed to chlorides. Then warm gently, dilute with 100 ml of water, heat to boiling, and after the silicic acid has settled, filter through a well fitted filter paper, Whatman No. 40. Wash the residue three or four times by decantation with hot 2 N hydrochloric acid, then transfer to the filter and wash with hot water until free from chloride. Place the precipitate in a platinum crucible and set aside for few minutes. The separation of the silicic acid is not quite complete; as much as 2 mg may remain in the filtrate. To remove this, once more evaporate the solution to dryness on the water-bath, and age in heat at 110°C for an hour, moisten the residue with 5 ml of concentrated hydrochloric acid, and allow to stand for not more than 15 minutes. Warm, dilute to 100 ml, heat to gentle boiling, and filter through a new and correspondingly small filter paper, washing with hot 2 N hydrochloric acid and with water as before. Reserve the filtrate and analyse it for ferric oxide, alumina, calcium oxide and magnesium oxide.

6.3.6 Combine the filter papers obtained in 6.3.5 and ignite them in a platinum crucible. Keep the temperature low until all the carbon is consumed, and do not allow the filter papers to catch fire. Finally, cover the crucible and ignite over a Meker burner, cool and weigh as impure silica.

6.3.7 The silica thus obtained is not absolutely pure. Moisten the impure silica with water, add a drop of concentrated sulphuric acid (specific gravity 1.18) and about 5 to 10 ml of pure hydrofluoric acid. Place the crucible in an air bath and evaporate under a hood until no more vapours are expelled. Remove the excess sulphuric acid by heating over a free flame. Raise the temperature gradually and finally heat the crucible over a Meker burner and again weigh.

6.3.8 Repeat the treatment with sulphuric and hydrofluoric acids, without adding any more water, until the contents of the crucible (usually ferric oxide and alumina) are at a constant weight. Deduct this amount from the weight of impure silica and add it to the precipitate obtained with the ammonia in the subsequent analysis.

6.4 Calculations — The percentage of silica shall be calculated by the following formula:

$$\text{Silica, percent} = \frac{\text{Weight in g of silica}}{\text{Weight in g of sample (W)}} \times 100$$

7. DETERMINATION OF FERROUS OXIDE BY VOLUMETRIC METHOD

7.1 Apparatus

7.1.1 Heater

7.1.2 Platinum Crucible — 30 ml capacity with lid.

7.1.3 Burette — 50 ml capacity.

7.1.4 Porcelain Basin and Wide Mouth Beaker

7.2 Reagents

7.2.1 Hydrofluoric Acid

7.2.2 Concentrated Sulphuric Acid — specific gravity 1.84.

7.2.3 Boric Acid Solution — saturated.

7.2.4 Potassium Permanganate Solution — standard, 1 ml is equivalent to 0.0056 g of iron.

7.3 Procedure — Take 0.5 g fibre from the laboratory sample in a 30 ml platinum crucible fitted with lid. Mix 10 ml of hydrofluoric acid and 10 ml of concentrated sulphuric acid and place the platinum crucible in a heater. At first heat slowly with the lid fully covering the crucible and then heat strongly avoiding turbulence of the liquid. After about 15 minutes, when the fibre is decomposed, take the platinum crucible out of the heater and cool in a ice-cold water-bath in a porcelain basin or in a wide mouthed beaker. After cooling, drop the crucible and the contents carefully into a wide mouthed flask containing 200 ml of cold (15°C) saturated boric acid solution. Titrate the cold mass with standard potassium permanganate solution.

7.4 Calculations — Determine ferrous oxide percentage by the following formula:

$$\text{Ferrous oxide, percent} = \frac{\text{Volume of potassium permanganate in ml} \times 0.0056 \times 1.28}{\text{Weight of sample in g}} \times 100$$

8. DETERMINATION OF FERRIC OXIDE

8.1 Apparatus

8.1.1 *Meker Burner*

8.1.2 *Platinum Crucible*

8.1.3 *Platinum Wire*

8.1.4 *Silica Crucible*

8.1.5 *Two Burettes* — each of 5 ml capacity.

8.1.6 *Filter Paper* — Whatman No. 40.

8.2 Reagents

8.2.1 *Bromine Water*

8.2.2 *Ammonium Chloride Solution* — 2 N.

8.2.3 *Ammonium Hydroxide Solution* — 2 N.

8.2.4 *Hydrochloric Acid Solution* — 2 N.

8.2.5 *Ammonium Nitrate* — 2 percent (w/v).

8.2.6 *Potassium Pyrosulphate* — solid.

8.2.7 *Dilute Hydrochloric Acid* — 1:1 (v/v).

8.2.8 *Stannous Chloride Solution* — Prepare by dissolving 15 g tin metal in 350 ml hot concentrated hydrochloric acid (specific gravity 1.19) and dilute to one litre.

8.2.9 *Mercuric Chloride Solution* — saturated.

8.2.10 *Diphenylamine Indicator* — Dissolve one gram in 100 ml sulphuric acid (specific gravity 1.84).

8.2.11 *Potassium Dichromate, Approximately 0.1 N* — Dissolve 4.9 g of potassium dichromate in water and dilute to one litre. Standardize against a known weight of pure iron using same method as in 8.3.

8.3 Procedure — Treat the filtrate obtained under 6.3.5 as indicated in 8.3.1 to 8.3.5.

8.3.1 Oxidize the iron back to the ferric state by adding bromine water (or a few drops of concentrated nitric acid) and boiling until the excess of the latter is expelled. Add 10 ml of 2 N ammonium chloride solution and several drops of methyl red indicator, and precipitate the aluminium and iron from the boiling hot solution by adding dilute ammonia free from carbonate until the solution turns yellow. Allow the precipitate to settle, filter and wash twice by decantation with hot water.

8.3.2 Redissolve by running hot 2 N hydrochloric acid through the filter paper into the beaker containing the greater part of the precipitate. Repeat the precipitation with ammonia as under **8.3.1**, and after filtering and washing by decantation, transfer the precipitate to the filter paper and wash with water containing 2 percent ammonium nitrate until free from chloride. Reserve the filtrate for calcium oxide and magnesium oxide determination.

8.3.3 Allow the precipitate to drain as completely as possible, and ignite wet in the crucible containing the residue obtained under **6.3.8**. After igniting strongly over a Meker burner, weigh the crucible; its contents represent the sum of alumina and ferric oxide.

NOTE — The percentage of total ferric oxide and alumina may be obtained by the following formula:

$$\text{Total alumina and ferric oxide, percent} = \frac{\text{Weight of alumina and ferric oxide contained in the crucible}}{\text{Weight of sample (W)}} \times 100$$

8.3.4 Fuse the mixed oxides with potassium pyrosulphate in a silica crucible using 15 to 20 times as much potassium. Before starting the fusion, heat the potassium in the silica crucible until fumes of sulphur trioxide evolve. Add the mixture of oxides to anhydrous potassium pyrosulphate and heat slowly with a small flame until complete fusion occurs. Add further small amount of additional pyrosulphate. When the fusion is complete, make a spiral from a platinum wire and insert in the melt. When solidified, remove by pulling the wire. Dissolve the melt in hydrochloric acid (1:1 by vol) stirring until all is dissolved. Dilute this solution to a definite volume and take one-half of it for the ferric oxide and the other half of it for the alumina determination.

8.3.5 Heat the liquid taken for the ferric oxide to about 80°C and while still hot reduce the ferric iron by adding stannous chloride drop by drop from a burette until the yellow colour of the ferric chloride just disappears, using a white piece of paper under the beaker for a

better background of colour change. Cool the solution to 15°C and, while stirring, add 10 ml of mercuric chloride solution. Formation of a light silky precipitate indicates that the conditions are correct. Stir the solution vigorously for one minute, add 3 drops of diphenylamine indicator solution and dilute to 150 to 200 ml with cold distilled water. Titrate with a standardized solution of potassium dichromate until the blue colour persists. The concentration of the potassium dichromate solution should be such that one millilitre of the standard solution is equivalent to 0.0056 g iron.

8.4 Calculations — Determine the ferric oxide percentage by the following formula:

$$\text{Ferric oxide, percent} = \frac{\text{Volume of potassium dichromate titer in ml} \times 0.0056 \times 2 \times 1.43}{\text{Weight of sample (IV)}} \times 100$$

9. DETERMINATION OF ALUMINIUM OXIDE

9.1 The percentage of aluminium oxide shall be obtained by the difference in the percentage of the total ferric oxide and aluminium oxide (see 8.3.3) and the percentage of ferric oxide (see 8.4).

10. DETERMINATION OF CALCIUM OXIDE BY OXALATE (GRAVIMETRIC) METHOD

10.1 Apparatus

10.1.1 Beaker — 600 ml capacity.

10.1.2 Glass Funnel

10.1.3 Platinum Crucible

10.1.4 Filter Paper — Whatman No. 40.

10.1.5 Meker Burner

10.1.6 Desiccator

10.2 Reagents

10.2.1 Hydrochloric Acid Solution — 3 N.

10.2.2 Ammonium Oxalate Solution — 0.5 N.

10.2.3 Ammonium Hydroxide Solution — 2 N.

10.3 Procedure

10.3.1 Make the filtrate, obtained from 8.3.2, acidic with hydrochloric acid. Bring to a volume of 400 ml, heat to 80° to 90°C, and slowly add, while stirring, 30 ml of hot 0.5 N ammonium oxalate solution. Slowly add ammonium hydroxide solution until the solution is slightly ammoniacal and allow the precipitate to stand several hours before filtering.

10.3.2 If considerable magnesium is present in the solution, some magnesium oxalate will come down with the calcium oxalate. Redissolve the calcium oxalate precipitate and repeat the precipitation, using a filter paper for filtering off the precipitate. When all the solution has passed through the filter paper, wash the precipitate with about 15 ml hot water. Then rinse the precipitate back into the original beaker by holding the funnel in an inverted position and directing a stream of hot water against it. Replace the funnel in the support and wash the filter paper with about 25 ml of hot 3 N hydrochloric acid. Heat the acid in a test tube, pour it upon the upper edge of the paper, and catch the liquid as it runs through the filter paper in the beaker containing the precipitate. Finally, wash the filter paper with a little hot water (and with dilute ammonia if it is to be used again for filtering the next precipitate).

10.3.3 Heat the dilute acid in the beaker and add a little more acid if necessary to dissolve the precipitate completely. Dilute the solution to about 250 ml and repeat the precipitation of the oxalate at the boiling temperature, adding ammonia and 5 ml more of the ammonium oxalate reagent.

10.3.4 Use a filter paper for collecting the precipitate. Wash the precipitate with hot water until free from chloride, and reserve the filtrate for the magnesium determination. Transfer the filter paper containing the precipitate to a weighed platinum crucible. Ignite carefully with the flame at the mouth of the crucible until the precipitate is dry, and then heat with a small flame at the base of the crucible until all the paper is decomposed without letting it take fire. Then gradually raise the temperature and heat over a Meker burner for an hour with the crucible in an upright position and covered. Cool to about 100°C, place in a desiccator, and weigh after 15 minutes. Repeat the heating until after cooling a constant weight is obtained. The calcium oxide is somewhat hygroscopic but is not difficult to weigh if it has

been washed free from chloride. Assume weight constant if it agrees within 0.2 mg with the previous weight.

10.4 Calculations — Determine calcium oxide percentage by the following formula:

$$\text{Calcium oxide, percent} = \frac{\text{Weight of calcium oxide}}{\text{Weight of sample (W)}} \times 100$$

11. DETERMINATION OF MAGNESIUM OXIDE

11.1 Apparatus

11.1.1 *Steam Bath or Hot Plate*

11.1.2 *Beaker and Watch-Glass*

11.1.3 *Filter Paper* — Whatman No. 40.

11.2 Reagents

11.2.1 *Concentrated Nitric Acid* — specific gravity 1.5.

11.2.2 *Concentrated Hydrochloric Acid* — specific gravity 1.16.

11.2.3 *Diammonium Hydrogen Phosphate* — solid.

11.2.4 *Phenolphthalein Indicator*

11.2.5 *Ammonium Hydroxide Solution* — 1.5 N.

11.2.6 *Concentrated Ammonium Hydroxide* — specific gravity 0.90.

11.2.7 *Ammonium Nitrate Solution* — saturated.

11.3 Procedure

11.3.1 Add 75 ml of concentrated nitric acid to the combined filtrate and washings reserved under 10.3.4, and evaporate to dryness on the steam bath or hot plate. Do not boil the solution or there will be loss by spattering. Keep the beaker covered with a watch-glass supported above the upper rim of the beaker. A glass triangle, or glass supports which are made for this purpose, should be used to support the watch-glass. To the small residue obtained, add 2 ml of concentrated hydrochloric acid and 25 ml of water. Heat nearly to boiling and, after a few minutes, filter off the silica residue through a small filter paper. Wash the beaker, and filter paper thoroughly with hot water. The silica comes from the action of reagents on reagent bottles or on the beaker used in the analysis.

11.3.2 Dilute the solution to about 150 ml; add about 1.2 g of diammonium hydrogen phosphate dissolved in a little water and a few drops of phenolphthalein indicator solution. Heat nearly to the boiling point, and then slowly add 1.5 N ammonium hydroxide solution until a faint pink colour is obtained and a slight precipitation takes place. Stir well for about a minute, touching the sides of the beaker as little as possible. When the precipitate has become distinctly crystalline, add more ammonium hydroxide solution until a deep colour is obtained with the phenolphthalein. Allow the solution to cool, then add one-fifth the solution's volume of concentrated ammonium hydroxide and allow to stand overnight.

11.3.3 Filter off the magnesium ammonium phosphate precipitate. After washing it with 1.5 N ammonium hydroxide solution moisten with a saturated solution of ammonium nitrate in 1.5 N ammonium hydroxide solution, dry and ignite very slowly and carefully at 1 000° to 1 100°C and weigh. The precipitate contains 36.21 percent magnesium oxide.

11.4 Calculations — Determine magnesium oxide percentage by the following formula:

$$\text{Magnesium oxide, percent} = \frac{\text{Weight of magnesium pyrophosphate (Mg}_2\text{P}_2\text{O}_7) \times 0.3621}{\text{Weight of sample (W)}} \times 100$$

12. DETERMINATION OF SODIUM OXIDE

12.1 Apparatus

12.1.1 *Platinum Dish and Platinum Crucible*

12.1.2 *Stout Platinum Wire*

12.1.3 *Radiator or Hot Plate*

12.1.4 *Water-Bath*

12.1.5 *Meker Burner*

12.1.6 *Mechanical Stirrer*

12.1.7 *Sintered Bed Glass or Porcelain Crucible — IG-4.*

12.1.8 *Drying Oven*

12.2 Reagents

12.2.1 *Dilute Sulphuric Acid — 1:5 (v/v).*

12.2.2 *Concentrated Hydrofluoric Acid — specific gravity 1.16.*

12.2.3 *Barium Hydroxide*

12.2.4 *Barium Chloride*

12.2.5 *Ammonium Carbonate*

12.2.6 *Ammonium Hydroxide*

12.2.7 *Hydrochloric Acid*

12.2.8 *Uranyl Acetate*

12.2.9 *Zinc Acetate*

12.2.10 *Sodium Zinc Uranyl Acetate*

12.2.11 *Absolute Alcohol*

12.2.12 *Ether or Acetone*

12.3 Procedure

12.3.1 Take one gram of the sample powder in a platinum crucible. Pour about 5 ml of dilute sulphuric acid (1:5, *v/v*), mix it with the powder by means of a glass rod or stout wire of platinum. Add 5 ml of concentrated hydrofluoric acid and evaporate until the sulphuric acid begins to escape. Repeat the evaporation with more hydrofluoric acid until the mineral is wholly decomposed. Then expel most of the sulphuric acid by heating over a radiator or hot plate. The expulsion of fluorine also should be thorough.

12.3.2 Heat the sulphate residue until fumes of sulphur trioxide entirely cease. Thus oxides or salts of iron, aluminium, titanium and phosphorus are rendered insoluble in water. Take the contents of the crucible with hot water in a beaker and wash the crucible thoroughly with hot water so that the transferring of the material from crucible to beaker is quantitative. Filter and wash the residue on the filter paper thoroughly with hot water four or five times. Reject the residue. Once a clear solution is obtained from the residue, precipitate the sulphate ion by barium chloride solution in slight excess. Without filtering, evaporate the liquid to dryness, take up with a little hot water and add alkali-free barium hydroxide solution until the solution is alkaline. In order to render the magnesium more insoluble again evaporate to dryness without filtering. Stir the residue up with water, filter and wash with a dilute alkali-free barium hydroxide solution. Warm the filtrate and precipitate most of the excess barium by ammonium hydroxide and ammonium carbonate. Repeat the operations until all the barium is removed. Then evaporate the final filtrate to dryness in a platinum dish and moisten the residue with a drop of hydrochloric acid. Evaporate until ammonia ceases to evolve, ignite very carefully till the residue just starts melting and weigh the alkali chlorides after cooling the dish in a desiccator which still contains a little magnesium as a basic chloride. If on dissolving of the chlorides

in a little water any residue is seen, filter and wash with water. Evaporate the filtrate to dryness and weigh as alkali chlorides.

12.3.3 Determination of Sodium as Sodium-Zinc Uranyl Acetate Hexahydrate — Add water to the residue obtained in 12.3.2 until the whole of it dissolves and evaporate on a water bath to about 1.5 ml. Add 15 ml of zinc uranyl acetate (prepared as indicated in 12.3.3.1) and stir vigorously, preferably mechanically, for at least 30 minutes. Allow to stand for one hour, and filter through a weighed sintered bed glass or porcelain filtering crucible. Wash the precipitate four times with 2-ml portions of the precipitating reagent (allow the wash liquid to drain completely before adding the next portion), then ten times with 95 percent alcohol saturated with sodium-zinc uranyl acetate at room temperature (2-ml portions) and finally with a little dry ether or acetone. Dry for 30 minutes only at 55° to 60°C. Weigh as sodium zinc uranyl acetate hexahydrate $[\text{NaZn}(\text{UO}_2)_2(\text{C}_2\text{H}_3\text{O}_2)_6 \cdot 6\text{H}_2\text{O}]$.

12.3.3.1 Preparation of zinc uranyl acetate — The reagent is prepared by mixing equal volumes of solutions A and B and filtering after standing overnight.

Solution A = Dissolve 20 g of crystallized uranyl acetate $[\text{UO}_2(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 2\text{H}_2\text{O}]$ in 4 ml of glacial acetic acid and 100 ml of water (warming may be necessary).

Solution B = Dissolve 60 g of crystallized zinc acetate $[\text{Zn}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 3\text{H}_2\text{O}]$ in 3 ml of glacial acetic acid and 100 ml of water.

12.4 Calculations — Determine the percentage of sodium oxide using the following formula:

$$\text{Sodium oxide, percent} = \frac{62}{1235.45} \times W \times 100$$

where

W = the weight of sodium-zinc uranyl acetate obtained from one gram of the sample.

13. DETERMINATION OF CARBON DIOXIDE FROM CARBONATES

13.1 Apparatus

13.1.1 Chemical Balance

13.1.2 Knorr Alkalimeter — consisting of the following parts, connected suitably. A typical arrangement of the above is given in Fig. 1.

- Gas washing bottle, A* — containing concentrated sulphuric acid which serves both to indicate the rate of flow and to prevent any water vapour entering the system from the atmosphere.

- b) *Drying tube or cylinder, B* — filled two-thirds with soda-asbestos absorbent (ascarite) and one-third with a drying agent to remove all carbon dioxide from the air.
- c) *Knorr alkalimeter unit, C* — which consists of dropping funnel, a distillation flask, and a condenser fitted with standard taper joints to form a unit assembly.
- d) *Gas washing bottle, D* — containing a solution of 5 to 10 percent by weight of silver sulphate in concentrated sulphuric acid. This serves to absorb any water vapour that escapes from the condenser and to remove any hydrochloric acid from the evolved gases.
- e) *Drying tube, E* — containing copper sulphate to absorb any hydrogen sulphide generated by the absorption of hydrochloric acid.
- f) *Drying tube, F* — filled with a suitable drying agent to effect complete drying.
- g) *Absorption tube, G* — filled two-thirds with soda-asbestos absorbent (ascarite) and one-third with a drying agent to absorb the carbon dioxide.
- h) *Absorption tube, H* — filled with a drying agent and soda-asbestos absorbent (ascarite) in the reverse direction to prevent carbon dioxide from entering the system.
- j) *Trap, J* — to prevent back-flow of water from the aspirator.
- k) *Valve or pinched tubing, K* — to control the rate of air flow pulled through the system by the aspirator.

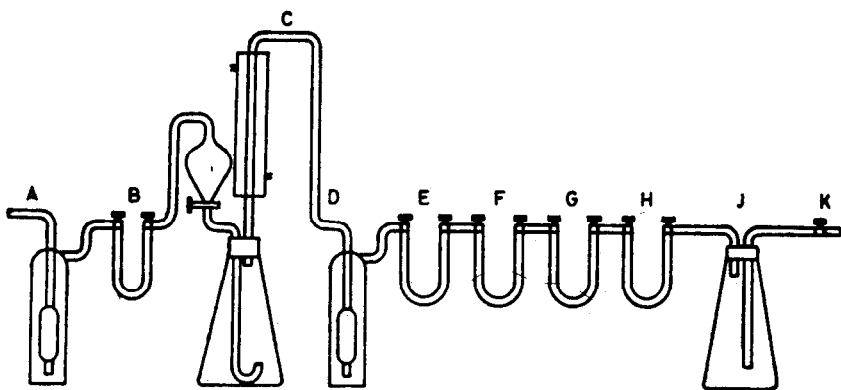


FIG. 1 KNORR ALKALIMETER FOR DETERMINATION OF CARBON DIOXIDE

13.2 Reagents

13.2.1 Concentrated Sulphuric Acid — specific gravity 1.84.

13.2.2 Soda-Asbestos Absorbent (Ascarite)

13.2.3 Silver Sulphate Solution — 10 percent by weight.

13.2.4 Copper Sulphate — solid.

13.2.5 Dilute Hydrochloric Acid — 1:1 (v/v).

13.3 Sampling — The sample shall be taken in the same manner as the laboratory test sample from which specimens were taken for the preceding analysis. A specimen shall be taken, weighing approximately one gram and dried to constant weight at 110°C weighing to the nearest 0.001 g. Let this sample weigh W grams.

13.4 Procedure

13.4.1 Check the gas-flow train to ensure freedom from leaks. Aspirate a current of air through the system at the rate of about two bubbles per minute for a period of 10 minutes. Stop the air current and remove the absorption tubes. Place the tubes within the balance case and allow it to stand for several minutes. When ready for weighing, open the stopcock momentarily and close. Weigh and repeat the above procedure. The second weight should agree with the first to within 0.005 g. If it does not, repeat this process until the two successive weighings agree. When a constant weight is reached, replace the absorption tubes in the gas train.

13.4.2 Transfer about one gram of the sample, weighed to the nearest 0.001 g, to the distillation flask. Wash down any adhering particles on the inside neck of the flask with distilled water. Add enough distilled water to the flask so when the apparatus is reconnected the tip of the dropping funnel will be submerged about 5 to 10 mm. Place 50 ml of hydrochloric acid (1:1) in the dropping funnel and replace the guard gas-absorption tube at the top of the funnel. Start the flow of water in the condenser and open all stopcocks except the one on the dropping funnel. Turn on the aspirator for medium suction. Adjust the stopcock on the dropping funnel so that the suction draws the acid slowly into the flask. When all the acid is in the flask, fully open the funnel stopcock and pull air through the system at the rate of two or three bubbles per second. After the reaction in the flask has subsided, warm the contents gently to boiling. When steam starts to condense in the condenser, turn off the heat. Continue to draw air through the apparatus for 30 minutes. Reweigh the absorption bulb as before. The gain in weight represents the carbon dioxide in the sample.

13.5 Calculations — Determine the carbon dioxide percentage by the following formula:

$$\text{Carbon dioxide, percent} = \frac{\text{Weight of carbon dioxide absorbed}}{\text{Weight of sample (W)}} \times 100$$

14. DETERMINATION OF WATER OF CRYSTALLIZATION

14.1 Apparatus

14.1.1 Tared Porcelain Crucible

14.1.2 Electric Furnace — $925^{\circ} \pm 25^{\circ}\text{C}$.

14.1.3 Desiccator

14.1.4 Chemical Balance

14.2 Procedure — Ignite in a tared porcelain crucible, the sample which has been dried to constant weight, by placing the crucible and contents in an electric furnace maintained at $925^{\circ} \pm 25^{\circ}\text{C}$ and continue the ignition for one hour. Cool in an oven at 110°C and then in a desiccator. Then reweigh to nearest 0.001 g.

14.3 Calculations — Determine the water of crystallization percentage by the following formula:

$$\text{Water of crystallization, percent} = \frac{\text{Loss on ignition, percent (see 14.2)}}{\text{Carbon dioxide, percent (see 13.5)}} \times 100$$

(Continued from page 2)

Panel for Amphibole Variety of Indigenous Asbestos, BDC 2: 3:1

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